ULTRASONIC EXTRACTION by EPA METHOD 3550C REVISION 3 (2007)

Facility Name:_____VELAP ID_____

Assessor Name:_____Analyst Name:_____Inspection Date_____

Relevant Aspect of Standards	Method Reference	Y	N	N/A	Comments
Records Examined: SOP Number/ Revision/ Date _____ Analyst:_____ Sample ID: _____ Date of Sample Preparation:_____ Date of Analysis:_____					
Were manufacturers' instructions followed regarding specific operational settings?	1.4				
Did analysts demonstrate abilities to produce acceptable results for the specific solvent systems and operating conditions for the analytes of interest at the concentrations of interest?	1.5 1.8 9.8				
Were method blanks determined to be free from interferences and contamination?	4.1 7.3				
Were chemicals used in this method of appropriate grades?	7.1 7.4				
Was anhydrous, granular sodium sulfate used purified by heating to 400^{o}C for 4 hours?	7.3				
Were IDPs for each sample preparation and determinative method combination done?	9.2				
Were IDPs done whenever a new or analyst was trained or significant changes to instrumentation were made?	9.2				
Were all glassware, equipment, and reagents demonstrated to be interference-free prior to any sample anlaysis?	9.3				
Did the extraction device have a minimum of 300 watts power?	11.0				
Did the extraction device have appropriately sized disrupter horns?	11.0				
Were horn tips tuned and maintained to manufacturer's instructions prior to use?	11.0				
Were horn tips inspected for wear prior to use?	11.0				
Were samples mixed with sodium sulfate, so that they formed free-flowing powders prior to solvent addition?	11.0 11.3.1.5				
Notes/Comments:					

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Were different extraction horns used for low concentration and high concentration protocols?	11.0				
Were three extractions used on low concentration samples?	11.0				
Did analysts observe active mixing of samples at some point after pulse activation?	11.0				
Were water layers decanted off of sediment/soil samples?	11.1.1				
Were multi-phase samples not extracted by this method?	11.1.2				
Were dry waste samples sieved or ground so that they would pass through a 1 mm sieve?	11.1.3				
Were gummy, fibrous, or oily samples reduced in size by some way to maximize surface area?	11.1.4				
When determinations were to be made on percent dry weight, were separate sample portions used for weight determinations?	11.2				
Low Concentration Extraction Procedure					
Was this procedure used when samples were expected to contain less than 20 mg/kg of organic analytes?	11.3				
Was approximately 30 g of sample weighed to the nearest 0.1 g used for this procedure?	11.3.1				
Were 1.0 mL volumes of surrogate and spike solutions added prior to addition of sodium sulfate?	11.3 11.3.1.2 11.3.1.3				
When gel permeation cleanup method 3640 was to be used, were twice the necessary volumes of spike and surrogate solutions added?	11.3.1.4				
Were steps performed quickly to minimize loss of volatile extractables?	11.3.1				
Were the ¾ inch disrupter horns placed about ½ inch below the surface of the solvent but above the sediment layer?	11.3.2				
Were the samples extracted ultrasonically for 3 minutes at full power or the manufacturer's recommended setting, on Pulse, and with the percent-duty knob a 50%?	11.3.3				
Notes/Comments:					

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Was the microtip probe **not** used?	11.3.3				
Were the extracts then decanted and filtered through a Whatmann No. 41 or equivalent filter paper?	11.3.4				
If extracts were not filtered, were they centrifuged at low speed to remove particles?	11.3.4				
Were the extraction processes repeated twice more with clean solvent?	11.3.5				
After the final extraction, were the samples and the three corresponding solvent rinses combined and filtered again?	11.3.5				
Medium/High Concentration Procedure					
Was this procedure used when more than 20 mg/kg of analytes were expected?	11.4				
Was approximately 2 g of sample weighed to the nearest 0.1 g used for this procedure?	11.4.1				
Were 1.0 mL volumes of surrogate and spiking solutions added the samples?	11.4.3				
When gel permeation cleanup method 3640 was to be used, were twice the necessary volumes of spike and surrogate solutions added?	11.4.4				
Were nonporous or wet samples mixed with 2 g of sodium sulfate?	11.4.5				
Was whatever volume of solvent necessary to bring the final volume to 10 mL added?	11.4.6				
Were the samples extracted with the 1/8 inch tapered microtip ultrasonic probe for 2 minutes with output control setting 5, the mode on Pulse, and the percent duty cycle at 50%?	11.4.7				
Were sample extracts filtered through 2-3 cm of glass wool?	11.4.8				
If the entirety of the extracts were not recovered in from the filtration, were the final sample calculations adjusted to account for the loss?	11.4.8				
Notes/Comments:					

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Relevant Aspect of Standards	**Method Reference**	**Y**	**N**	**N/A**	**Comments**
Kuderna-Danish (KD) Concentration Technique					
Was this procedure used when necessary to meet instrument sensitivity requirements?	11.5				
Were extracts dried prior to concentration by filtering through approximately 10 g of anhydrous sodium sulfate?	11.5.2				
Were collection tubes and drying columns rinsed with additional solvent after filtration to achieve full transfer?	11.5.3				
Was the water bath temperature about 15-20^{o}C?	11.5.4				
When the extract volumes reached 1 mL, were the K-D apparatuses removed from the water bath and cooled for at least 10 minutes?	11.5.4				
Was extract prevented from evaporating to dryness?	11.5.4				
Was solvent exchange, if necessary, done by adding new solvent to 1 mL extract volume and repeating concentration?	11.5.4.1				
Was the apparatus rinsed with 1-2 mL of solvent and reconcentrated to achieve transfer?	11.5.5				
If micro-snyder column technique was used, were extracts evaporated to 0.5 mL followed by rinsing apparatus and bringing extract volume back up to 1.0-2.0 mL?	11.6.1.1				
When Nitrogen evaporation technique was used, were the sample extracts placed in water baths of about 30^{o}C and evaporated with clean, dry nitrogen?	11.6.2.1				
Were concentrator walls rinsed down several times during concentration by nitrogen evaporation?	11.6.2.2				
Notes/Comments:					

TABLE 1

EXAMPLE EFFICIENCIES OF VARIOUS EXTRACTION SOLVENT SYSTEMS FOR SELECTED COMPOUNDS[a]

			Solvent System[d]									
			A		B		C		D		E	
Compound	CAS No.[b]	ABN[c]	%R	SD	%R	SD	%R	SD	%R	SD	%R	SD
4-Bromophenyl phenyl ether	101-55-3	N	64.2	6.5	56.4	0.5	86.7	1.9	84.5	0.4	73.4	1.0
4-Chloro-3-methylphenol	59-50-7	A	66.7	6.4	74.3	2.8	97.4	3.4	89.4	3.8	84.1	1.6
Bis(2-chloroethoxy)methane	111-91-1	N	71.2	4.5	58.3	5.4	69.3	2.4	74.8	4.3	37.5	5.8
Bis(2-chloroethyl) ether	111-44-4	N	42.0	4.8	17.2	3.1	41.2	8.4	61.3	11.7	4.8	1.0
2-Chloronaphthalene	91-58-7	N	86.4	8.8	78.9	3.2	100.8	3.2	83.0	4.6	57.0	2.2
4-Chlorophenyl phenyl ether	7005-72-3	N	68.2	8.1	63.0	2.5	96.6	2.5	80.7	1.0	67.8	1.0
1,2-Dichlorobenzene	95-50-1	N	33.3	4.5	15.8	2.0	27.8	6.5	53.2	10.1	2.0	1.2
1,3-Dichlorobenzene	541-73-1	N	29.3	4.8	12.7	1.7	20.5	6.2	46.8	10.5	0.6	0.6
Diethyl phthalate	84-66-2	N	24.8	1.6	23.3	0.3	121.1	3.3	99.0	4.5	94.8	2.9
4,6-Dinitro-*o*-cresol	534-52-1	A	66.1	8.0	63.8	2.5	74.2	3.5	55.2	5.6	63.4	2.0
2,4-Dinitrotoluene	121-14-2	N	68.9	1.6	65.6	4.9	85.6	1.7	68.4	3.0	64.9	2.3
2,6-Dinitrotoluene	606-20-2	N	70.0	7.6	68.3	0.7	88.3	4.0	65.2	2.0	59.8	0.8
Heptachlor epoxide	1024-57-3	N	65.5	7.8	58.7	1.0	86.7	1.0	84.8	2.5	77.0	0.7
Hexachlorobenzene	118-74-1	N	62.1	8.8	56.5	1.2	95.8	2.5	89.3	1.2	78.1	4.4
Hexachlorobutadiene	87-68-3	N	55.8	8.3	41.0	2.7	63.4	4.1	76.9	8.4	12.5	4.6
Hexachlorocyclopentadiene	77-47-4	N	26.8	3.3	19.3	1.8	35.5	6.5	46.6	4.7	9.2	1.7
Hexachloroethane	67-72-1	N	28.4	3.8	15.5	1.6	31.1	7.4	57.9	10.4	1.4	1.2
5-Nitro-*o*-toluidine	99-55-8	B	52.6	26.7	64.6	4.7	74.7	4.7	27.9	4.0	34.0	4.0
Nitrobenzene	98-95-3	N	59.8	7.0	38.7	5.5	46.9	6.3	60.6	6.3	13.6	3.2
Phenol	108-95-2	A	51.6	2.4	52.0	3.3	65.6	3.4	65.5	2.1	50.0	8.1
1,2,4-Trichlorobenzene	120-82-1	N	66.7	5.5	49.9	4.0	73.4	3.6	84.0	7.0	20.0	3.2

Footnotes appear on the following page.

TABLE 2

SPECIFIC EXTRACTION CONDITIONS FOR VARIOUS DETERMINATIVE METHODS

Determinative Method	Recommended Solvent for Analysis	Recommended Solvent for Cleanup	Extract Volume Recommended for Cleanup (mL)	Recommended Final Extract Volume for Analysis (mL)[a]
8041	2-propanol	hexane	1.0	1.0, 0.5[b]
8061	hexane	hexane	2.0	10.0
8070	methanol	methylene chloride	2.0	10.0
8081	hexane	hexane	10.0	10.0
8082	hexane	hexane	10.0	10.0
8085	isooctane	hexane	10.0	NS
8091	hexane	hexane	2.0	1.0
8100	none	cyclohexane	2.0	1.0
8111	hexane	hexane	2.0	10.0
8121	hexane	hexane	2.0	1.0
8141	hexane	hexane	10.0	10.0
8270[c]	none	-	-	1.0
8310	acetonitrile	-	-	1.0
8321	methanol	-	-	1.0
8325	methanol	-	-	1.0
8410	methylene chloride	methylene chloride	10.0	0.0 (dry)

[a] These volumes are only recommendations. The final extract volume should be established based on the sensitivity necessary for the intended application. For methods where the recommended final extract volume is 10.0 mL, the volume may be reduced to as low as 1.0 mL to achieve lower limits of quantitation.

[b] Phenols may be analyzed by Method 8041, using a 1.0-mL 2-propanol extract by GC/FID. Method 8041 also contains an optional derivatization procedure for phenols which results in a 0.5-mL hexane extract to be analyzed by GC/ECD.

[c] The specificity of GC/MS may make cleanup of the extracts unnecessary. Refer to Method 3600 for guidance on the available cleanup procedures, if necessary.

NS = Not specified. The final extract volume should be established based on the sensitivity necessary for the intended application.